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Thermal Conductivities of Ionic Liquids over the Temperature Range from 293 K to 353 K

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The thermal conductivities of 11 ionic liquids were determined, over the temperature range from 293 K to 353 K, at atmospheric pressure, using an apparatus based on the transient hot-wire method. For each of the ionic liquids studied, the thermal conductivities were found to be between (0.1 and 0.2) $W{\cdot}m^{-1}{\cdot}K^{-1}$, with a slight decrease observed on increasing temperature. The uncertainty is estimated to be less than $\pm$ 0.002 $W{\cdot}m^{-1}{\cdot}K^{-1}$. In all cases, a linear equation was found to give a good fit to the data. The effects of water content and chloride content on the thermal conductivities of some of the ionic liquids were investigated. In each case, the thermal conductivities of the water + ionic liquid and chloride + ionic liquid binary mixtures were found to be less than the weighted average of the pure component thermal conductivities. This effect was adequately modeled using the Jamieson correlation. Chloride contamination at typical postsynthesis levels was found to have no significant effect on the thermal conductivities of the ionic liquid studied.

Introduction

Ever-increasing attention is being given to ionic liquids as novel solvents for a wide range of chemical reactions. This level of interest stems mainly from the unique properties of such liquids, including the ability to tune their physical and chemical properties by varying the cation−anion combinations, their wide liquid temperature range, and their ability to simultaneously dissolve inorganic homogeneous catalysts and organic reagents. In addition, due to their negligible vapor pressures, ionic liquids are often cited as potential environmentally benign solvents to replace volatile organic compounds (VOCs) in industrial chemical processes.[1] However, although the phase equilibria,[2] the electrochemistry,[3] and a wide range of chemical reactions[4−8] have been extensively studied in ionic liquids, there still exists a shortage of the physical property data required for the engineering design of industrial scale processes. In particular, only a few data exist in the literature for the thermal conductivities of ionic liquids,[9,10] yet this information is essential for the safe and efficient design of heat transfer equipment such as chemical reactors and heat exchangers.[11] For example, when designing such process equipment, the convective heat transfer coefficient, which is a measure of the rate at which heat is transferred between a fluid and its surroundings by convection, must be determined. This design parameter is usually calculated from the physical properties of the fluids using correlations developed for different geometric arrangements. Thermal conductivity is one of the physical properties having the greatest influence on the convective heat transfer coefficient; therefore, it is essential that it is known for all fluids involved in the process.

In this study, the thermal conductivities of a range of commonly used ionic liquids have been determined, at ambient pressure, in the temperature range from 293 K to 353 K. The ionic liquids studied include 1-alkyl-3-methylimidazolium bis(trifluoromethylsulfonyl)imides ($[C_n\text{mim}][NTf_2]$), for $n = 2$, 4, 6, 8, and 10, 1-butyl-3-methylimidazolium trifluoromethanesulfonate ($[C_4\text{mim}][OTf]$), 1-ethyl-3-methylimidazolium ethylsulfate ($[C_2\text{mim}][EtSO_4]$), trihexyl(tetradecyl)phosphonium chloride ($[(C_6H_{13})_3P(C_{14}H_{29})]Cl$), trihexyl(tetradecyl)phosphonium bis(trifluoromethylsulfonyl)imide ($[(C_6H_{13})_3P(C_{14}H_{29})][NTf_2]$), 1-butyl-1-methylpyrrolidinium tris(pentafluoroethyl)trifluorophosphate ($[C_4\text{mpyrr}][FAP]$), and 1-butyl-1-methylpyrrolidinium bis(trifluoromethylsulfonyl)imide ($[C_4\text{mpyrr}][NTf_2]$). This temperature range was chosen as it is applicable to the temperature conditions used for many of the chemical reactions previously conducted in such ionic liquids. Previous studies have shown that the physical properties of ionic liquids can be greatly affected by the presence of chloride ions and/or water.[12] Many ionic liquids are prepared via an ion exchange process to convert the halide-based ionic liquid to the desired anion-based material. Although the subsequent halide contamination in the ionic liquid may be greatly reduced by washing with water, residual amounts are always present in the final ionic liquid, and therefore, the effect of chloride content on the thermal conductivity of $[C_6\text{mim}][NTf_2]$ has been studied. Furthermore, because all ionic liquids are hygroscopic coupled with the obvious difficulties in tightly controlling water content within an industrial environment, the effect of the presence of water on the thermal conductivities of $[C_4\text{mim}][OTf]$ and $[C_2\text{mim}][EtSO_4]$ has also been investigated.

Experimental

Materials. All ionic liquids were prepared in-house, except for $[(C_6H_{13})_3P(C_{14}H_{29})]Cl$, which was obtained from Cytec under the brand name CYPHOS IL 101, and $[C_4\text{mpyrr}][FAP]$, which was obtained from Merck. The $[C_n\text{mim}][NTf_2]$ range of ionic liquids was prepared by metathesis reaction of the appropriate $[C_n\text{mim}]Cl$ with $Li[NTf_2]$ according to previously reported procedures.[13] An analogous procedure was used to prepare $[(C_6H_{13})_3P(C_{14}H_{29})][NTf_2]$ from $[(C_6H_{13})_3P(C_{14}H_{29})]Cl$. $[C_4\text{mim}][OTf]$ was prepared by metathesis reaction of $[C_4\text{mim}]Cl$ with $Li[OTf]$ according to previously reported procedures.[14] $[C_2\text{mim}][EtSO_4]$ was prepared by reacting 1-methylimidazole

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with diethyl sulfate according to previously reported procedures.[15] Samples were washed 5 to 6 times using distilled water to ensure removal of any remaining lithium chloride byproduct or starting materials and dried overnight at 70 °C under a high vacuum (0.1 Pa) prior to use. All ionic liquids were analyzed by ^{1}H and ^{13}C NMR and elemental analysis and showed excellent agreement with the reported data. In addition, the water content using Karl−Fischer titration and chloride content using ion chromatography were measured prior to the measurements.[16] In all cases, the water mass fraction was found to be less than 0.0008, and the chloride mass fraction was less than $5 \cdot 10^{-6}$, unless otherwise stated.

Equipment and Procedure. Thermal conductivity data were measured using a KD2 Pro Thermal Properties Analyzer (Labcell Ltd., UK). The principle of measurement is based on the transient hot-wire method.[17] The meter consists of a thermal probe (1.3 mm diameter, 60 mm length), containing a heating element and a thermoresistor, which should be inserted into the sample vertically, rather than horizontally, to minimize the possibility of inducing convection. The measurement is made by heating the probe within the sample while simultaneously monitoring the temperature change of the probe. A microprocessor, connected to the probe, is used to control the heating rate, measure the temperature change data, and calculate the thermal conductivity based on a parameter-corrected version of the temperature model given by Carslaw and Jaeger[18] for an infinite line heat source with constant heat output and zero mass, in an infinite medium. For this model to accurately describe the physical behavior of a system, the heat source must closely approximate an infinitely long, thin line. Kuitenberg et al.[19] describe the solutions for a heated cylindrical source with a non-negligible radius and finite length which is used for this probe. Both models give equally good fit to the temperature data[20] but differ slightly in value for the fitting parameters, and these differences can be accounted for by the calibration, allowing the former, more simple model to be used which has been employed herein.

Approximately 50 cm^3 of the sample to be analyzed was sealed in a glass sample vial, and the probe was inserted vertically into the sample via a purpose-made port in the lid of the vial. The sealed vial was then fully immersed in a temperature-controlled water bath (Grant GD120) and allowed to equilibrate at the desired temperature. Once the sample reached the required temperature, a further 15 min was allowed to pass before carrying out the measurement to ensure complete equilibration. At least four measurements were taken at each temperature, with a delay of at least 15 min between each measurement, to ensure reproducibility. Before and after analysis of the ionic liquid samples, the meter was calibrated using water and a standard sample of glycerol of known thermal conductivity. Although it would have been preferable to also calibrate the equipment using a liquid of a lower thermal conductivity than the ionic liquids, such liquids also tend to have very low viscosities and are susceptible to errors due to convective currents. Therefore, it should be noted that because the calibration is an extrapolation rather than an interpolation there may be some systematic error that has not been accounted for. The uncertainty of the thermal conductivity was estimated, from the standard deviations of experimental and calibration measurements, to be ± 0.005 $W \cdot m^{-1} \cdot K^{-1}$. The uncertainty of the temperature is estimated to be ± 1 K.

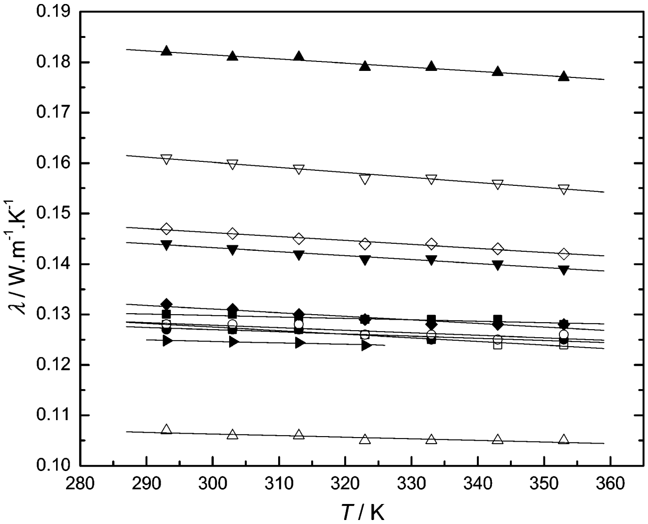

Figure 1. Thermal conductivities of ionic liquids as a function of temperature: ■, [C_2mim][NTf_2]; □, [C_4mim][NTf_2]; ●, [C_6mim][NTf_2]; ○, [C_8mim][NTf_2]; ◆, [C_{10}mim][NTf_2]; ◇, [C_4mim][OTf]; ▲, [C_2mim]-[$EtSO_4$]; △, [C_4mpyrr][FAP]; solid arrow pointing right, [C_4mpyrr][NTf_2]; ▼, [$(C_6H_{13})_3P(C_{14}H_{29})$][$NTf_2$]; ▽, [$(C_6H_{13})_3P(C_{14}H_{29})$]Cl. Lines correspond to the fit of the data using eq 1. The fitted parameters are given in Table 3.

Table 1. Thermal Conductivities, λ, of [$C_n$mim][NTf_2] Ionic Liquids for $n = 2$, 4, 6, 8, and 10, in the Temperature Range from 293 K to 353 K

	λ/$W \cdot m^{-1} \cdot K^{-1}$				
T/K	$n = 2$	$n = 4$	$n = 6$	$n = 8$	$n = 10$
293	0.130	0.128	0.127	0.128	0.132
303	0.130	0.127	0.127	0.128	0.131
313	0.129	0.127	0.127	0.128	0.130
323	0.129	0.126	0.126	0.126	0.129
333	0.129	0.125	0.125	0.126	0.128
343	0.129	0.124	0.125	0.125	0.128
353	0.128	0.124	0.125	0.126	0.128

Results and Discussion

The thermal conductivity data for the 11 ionic liquids studied over the temperature range from 293 K to 353 K are given in Figure 1 and summarized in Table 1, for the [$C_n$mim][NTf_2] ionic liquids, and in Table 2, for the other ionic liquids. All of the ionic liquids studied had similar thermal conductivities, each within the range 0.1 $W \cdot m^{-1} \cdot K^{-1}$ to 0.2 $W \cdot m^{-1} \cdot K^{-1}$. Increasing temperature was found to cause the thermal conductivities of each ionic liquid to decrease slightly, with a linear dependence observed over the temperature range investigated. Varying the alkyl chain length, n, of the [$C_n$mim][NTf_2] ionic liquids had no significant effect on the thermal conductivity. However, changing the cation type resulted in a larger variation as shown by the difference between [C_4mim][NTf_2], with a thermal conductivity of 0.128 $W \cdot m^{-1} \cdot K^{-1}$, and [$(C_6H_{13})_3P(C_{14}H_{29})$]-[$NTf_2$], with a value of 0.144 $W \cdot m^{-1} \cdot K^{-1}$, at 293 K. The anion also had a significant effect on the thermal conductivity; for example, replacing the anion from [NTf_2]$^-$ to [$EtSO_4$]$^-$ caused the thermal conductivity to increase from 0.130 $W \cdot m^{-1} \cdot K^{-1}$ to 0.182 $W \cdot m^{-1} \cdot K^{-1}$, at 303 K. Of all the ionic liquids studied, [C_4mpyrr][NTf_2] had the lowest thermal conductivity, with a value of 0.107 $W \cdot m^{-1} \cdot K^{-1}$ at 293 K. Again, the influence of the anion is clear because the thermal conductivity increases to 0.125 $W \cdot m^{-1} \cdot K^{-1}$ when the anion is changed to [NTf_2]$^-$.

The thermal conductivity data for the ionic liquids are compared with those of a range of some commonly used molecular solvents[21] in Figure 2. Similar values were found for the ionic liquids and toluene, methanol, and ethylene glycol, whereas water showed a significantly higher thermal conductivity than the ionic liquids studied. It should be noted that convection effects were not thought to be significant in the case

Table 2. Thermal Conductivities, λ, of Various Ionic Liquids in the Temperature Range from 293 K to 353 K

	λ/W·m^{-1}·K^{-1}					
T/K	[C$_4$mim][OTf]	[C$_2$mim][EtSO$_4$]	[C$_4$mpyrr][FAP]	[C$_4$mpyrr][NTf$_2$]	[(C$_6$H$_{13}$)$_3$P(C$_{14}$H$_{29}$)][NTf$_2$]	[(C$_6$H$_{13}$)$_3$P(C$_{14}$H$_{29}$)]Cl
293	0.147	0.182	0.107	0.125	0.144	0.161
303	0.146	0.181	0.106	0.125	0.143	0.160
313	0.145	0.181	0.106	0.124	0.142	0.159
323	0.144	0.179	0.105	0.124	0.141	0.157
333	0.144	0.179	0.105	–	0.141	0.157
343	0.143	0.178	0.105	–	0.140	0.156
353	0.142	0.177	0.105	–	0.139	0.155

of the ionic liquid measurements. In general, convection is found to lead to higher than expected thermal conductivities which increase with temperature due to the decrease in viscosity leading to greater free convection.[22] However, the thermal conductivity of the ionic liquids was found to decrease with increasing temperature, thus suggesting that convection was not influencing the values obtained.

Various attempts have been made to derive theoretical equations to explain observed values of thermal conductivities in liquids, with many giving some qualitative agreement with experimentally observed results.[23] In most cases, it is assumed that heat is conducted through a liquid via longitudinal oscillations and that the thermal conductivity of the liquid decreases as the average distance between the centers of molecules is increased. Although the ionic liquids studied all have significantly higher densities than most molecular liquids,[24] this is offset by the large size of their ions. Neutron diffraction and theoretical studies of imidazolium-based ionic liquids have shown that the distance between the center of the cation and anion is typically >4 Å[25] resulting in the low thermal conductivities compared with water, for example.

Figure 3 shows how the results obtained in this study compare with the experimental thermal conductivity data previously reported for ionic liquids. Direct comparisons can only be made with two experimental values presented by Frez et al.[10] Therein, a transient grating technique was used to determine thermal conductivities of [C$_2$mim][NTf$_2$] at 295 K and [C$_4$mim][NTf$_2$] at 298 K, with results of 0.12 W·m^{-1}·K^{-1} and 0.108 W·m^{-1}·K^{-1}, respectively. In each case, the results are lower by 10 % for [C$_2$mim][NTf$_2$] and by 20 % for [C$_4$mim][NTf$_2$] compared with the measurements from the present study. This discrepancy may be due to the fact that the transient grating technique does not directly measure thermal conductivity but measures the thermal diffusivity. The thermal conductivity, in this case, is then calculated from the measured thermal diffusivity and previously reported values for heat capacity and density which may lead to increased errors from the multiple measurements. This is particularly true if the same sample of ionic liquid is not used for each value, as the value depends strongly on the purity of the ionic liquid used. The transient hot-wire method, used by Van Valkenburg et al.[9] and in the current study, is a direct and absolute method of determining thermal conductivity, and therefore, sources of experimental error can more easily be determined and minimized. A similar discrepancy is found between the thermal conductivity of [C$_4$mim][BF$_4$] reported by Van Valkenburg et al.[9] and Frez et al.[10] As found herein, the transient grating technique gave a value which was around 15 % lower than when the transient hot-wire method was employed. The general trends observed in this study, such as the effects of changing temperature or anion, are very similar to those observed by Van Valkenburg et al.[9] Importantly, the results for [C$_3$dmim][NTf$_2$] agree well with the data for [C$_n$mim][NTf$_2$]-based ionic liquids reported herein.

For each of the ionic liquids studied, the thermal conductivity was found to be only weakly dependent on temperature, and the data could be fitted with a linear correlation of the form

$$\lambda = aT + b \tag{1}$$

Table 3 summarizes the fitted parameters, a and b, for each of the ionic liquids studied. This type of simple correlation was also applied by Van Valkenburg et al.[9] for various ionic liquids and is very useful for engineering calculations involving heat

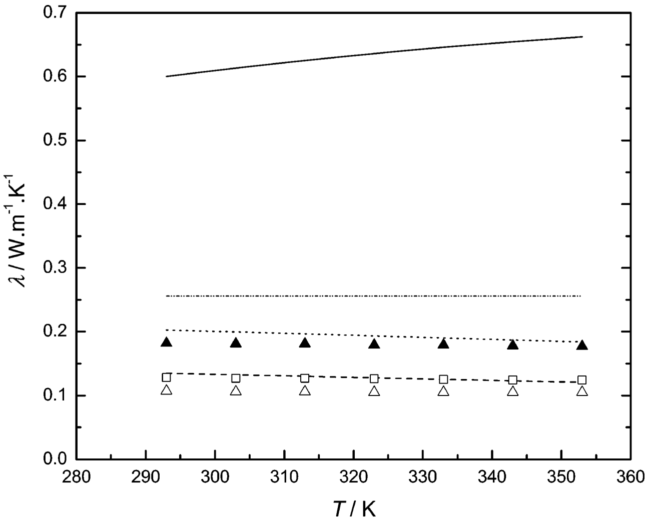

Figure 2. Comparison of thermal conductivity, k, against temperature between some ionic liquids (▲, [C$_2$mim][EtSO$_4$]; □, [C$_4$mim][NTf$_2$]; △, [C$_4$mpyrr][FAP]) and literature correlations[21] for molecular liquids (____, water; _ . _ . _, ethylene glycol; ...., methanol; and _ _ _ _, toluene).

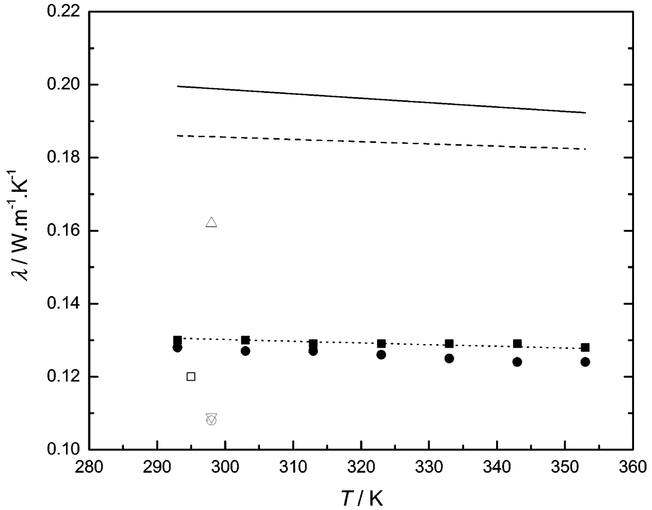

Figure 3. Comparison of plots of thermal conductivity, λ, against temperature between data from this study (■, [C$_2$mim][NTf$_2$]; ●, [C$_4$mim]-[NTf$_2$]) and other literature data for ionic liquids (Van Valkenburg et al.[9] correlations: ____, [C$_2$mim][BF$_4$]; _ _ _, [C$_4$mim][BF$_4$]; ...., [C$_3$dmim]-[NTf$_2$] and Frez et al.: □, [C$_2$mim][NTf$_2$]; ○, [C$_4$mim][NTf$_2$]; △, [C$_4$mim]-[BF$_4$]; ▽, [C$_4$mim][PF$_6$]).

Table 3. Parameters Obtained by Fitting Equation 1 to Thermal Conductivity vs Temperature Data for Various Ionic Liquids

ionic liquid	$10^5\,a$ / $W{\cdot}m^{-1}{\cdot}K^{-2}$	b / $W{\cdot}m^{-1}{\cdot}K^{-1}$
[C_2mim][NTf_2]	−2.857	0.1384
[C_4mim][NTf_2]	−7.143	0.1489
[C_6mim][NTf_2]	−4.285	0.1398
[C_8mim][NTf_2]	−5.000	0.1429
[C_{10}mim][NTf_2]	−7.143	0.1525
[C_4mim][OTf]	−7.857	0.1698
[C_2mim][$EtSO_4$]	−8.214	0.2061
[C_4mpyrr][FAP]	−3.214	0.1160
[C_4mpyrr][NTf_2]	−2.900	0.1334
[$(C_6H_{13})_3P(C_{14}H_{29})$][$NTf_2$]	−7.857	0.1668
[$(C_6H_{13})_3P(C_{14}H_{29})$]Cl	−10.000	0.1902

Table 4. Effect of the Mass Fraction of Component 2, w_2, on the Thermal Conductivities, λ, of the Binary Mixtures [C_4mim][OTf] (1) + Water (2), [C_2mim][$EtSO_4$] (1) + Water (2), and [C_6mim][NTf_2] (1) + [C_6mim]Cl (2) at 293 K

	$\lambda/W{\cdot}m^{-1}{\cdot}K^{-1}$		
w_2	[C_4mim][OTf] + water	[C_2mim][$EtSO_4$] + water	[C_6mim][NTf_2] + [C_6mim]Cl
0	0.147	0.181	0.128
0.01	0.150	0.181	0.128
0.05	0.157	0.190	0.129
0.10	0.173	0.201	0.130
0.20	0.221	0.232	0.132
0.50	0.356	0.352	0.139
0.70	0.440	0.445	0.151
1.00	0.607	0.607	0.172

transfer over a range of temperatures, particularly for the use of process simulation packages.

The effect of water content on the thermal conductivities of [C_4mim][OTf] and [C_2mim][$EtSO_4$] was studied, at 293 K, with the results shown in Table 4 and plotted in Figure 4. The addition of small amounts of water, up to a mass fraction of 0.01, had no significant effect on the ionic liquid thermal conductivity; however, above this mass fraction, the thermal conductivity of the mixture increased but was always less than the mass fraction average of the thermal conductivities of the pure components. This phenomenon is also commonly found for mixtures of molecular liquids and can be fitted with the Jamieson correlation (2):[26]

$$\lambda_m = w_1\lambda_1 + w_2\lambda_2 - \alpha(\lambda_2 - \lambda_1)[1 - (w_2)^{1/2}]w_2 \qquad (2)$$

where w_1 and w_2 are mass fractions; α is an adjustable parameter; and the components are selected so that $\lambda_2 > \lambda_1$. The solid lines in Figure 4 represent this correlation when fitted to the experimental data; α values of 0.4211 and 0.7043 were obtained for the binary mixtures [C_4mim][OTf] (1) + water (2) and [C_2mim][$EtSO_4$] (1) + water (2), respectively.

The effect of chloride content on the thermal conductivity of [C_6mim][NTf_2] was also investigated. Table 4 and Figure 4 also show the results following the addition of [C_6mim]Cl. As found with water, below a mass fraction of 0.05, no significant change in the thermal conductivity was observed. Above this mass fraction, the thermal conductivity of the binary mixture was found to increase but remained below the mass fraction average of the pure component values. The Jamieson correlation was also fitted to this data, with a value of $\alpha = 1.393$ being obtained for the fitted parameter. As an excellent fit was observed for both ionic liquid−ionic liquid and water−ionic liquid mixtures, this suggests that, although developed for molecular liquids, this is a general correlation for a wide range of liquid mixtures.

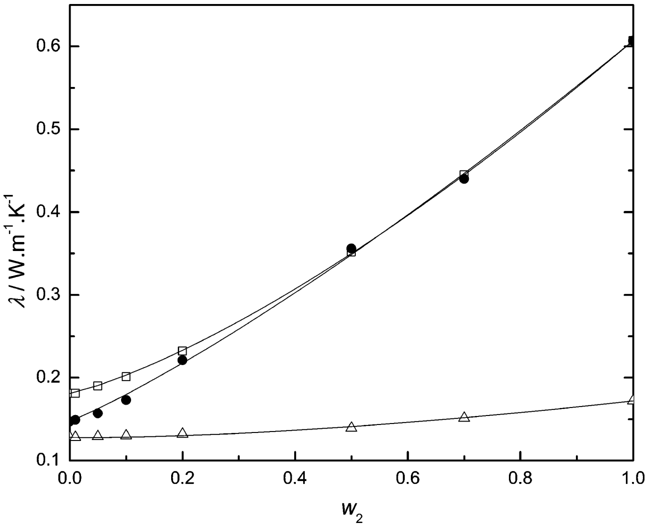

Figure 4. Plot showing the effect of the mass fraction of component 2, w_2, on the thermal conductivities, k, of the binary mixturesat 293 K: ●, [C_4mim][OTf] (1) + water (2); ■, [C_2mim][$EtSO_4$] (1) + water (2); and △, [C_6mim][NTf_2] (1) + [C_6mim]Cl (2). Solid lines represent the fit of the data by eq 2.

Conclusions

The thermal conductivities of a range of ionic liquids have been determined over the temperature range from 293 K to 353 K. In general, the thermal conductivity behavior of ionic liquids was found to be similar to organic molecular liquids, such as methanol or toluene, and much lower than that reported for water. Mole fractions of water up to 0.01 and mole fractions of chloride up to 0.05 have been shown to have little effect on the thermal conductivity of those ionic liquids studied; however, beyond these mole fractions, both contaminants caused thermal conductivity to increase. Water−ionic liquid and ionic liquid−ionic liquid binary systems were found to have thermal conductivities lower than the weighted average of the thermal conductivities of the pure components. The Jamieson correlation was found to adequately model the binary data.

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